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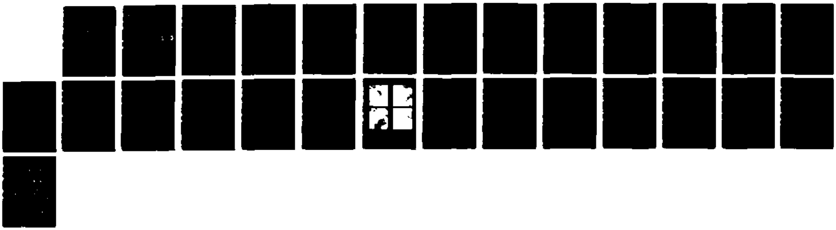

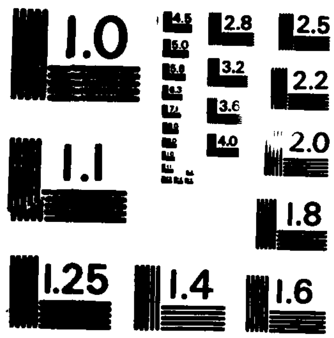

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ADVANCED PROCESSING AND PROPERTIES OF HIGH PERFORMANCE ALLOYS

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Progress is reviewed for a research program whose purpose is to provide a fundamental understanding of the applications and consequences of selected advanced processing techniques to high performance alloys. The research ranges from modeling studies of the fracture of alloys containing porosity to the high temperature deformation of rapidly solidified, dispersion-strengthened titanium alloys. Many aspects of the research are also fundamental studies of fracture utilizing engineering alloys containing processing-induced defects.

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Progress for the period October 1, 1984 to September 30, 1985 is reviewed for the following studies comprising this research program:

(1) the influence of void/pore distributions on ductile fracture;
(2) the effect of porosity on low cycle fatigue;
(3) hot isostatic pressing of metallic powders;
(4) deformation of rapidly solidified Ti alloys at elevated temperatures; and
(5) the combined effect of stress state and grain size on hydride-induced hydrogen embrittlement. . Keywords: powder metallurgy.

INTRODUCTION

High performance alloys are used primarily in applications requiring high strength and good fracture resistance over a range of temperatures and in-service environments. Expanding the use of these alloys is often critical to a desired increase in the performance or payload of a system. In recent years, advances in properties of such alloys have been limited to small, incremental improvements, while the high cost of a finished part has also confined their application to selected critical components. This has resulted in an increased application of advanced processing techniques to high performance alloys both to create new alloys exhibiting large property improvements and/or to reduce the cost of finished component by using near net-shape fabrication methods. One example is application of rapid solidification processing as a method of developing new families of high performance alloys.[1-4] Fabrication of rapidly solidified alloys requires extensive processing, which is usually based on powder metallurgy techniques. It should be noted that powder metallurgy is also being used as a new method of reducing component cost in fabricating complex-shaped components from high performance alloys.[5,6]

An underlying problem in high performance alloy technology is that the rate of applying the new processing methods to these alloys exceeds the development of the basic understanding necessary to predict the resulting component behavior in service. Thus, service reliability may be impaired. A specific concern is that these materials can contain processing-induced defects, such as porosity or inclusions, which can seriously degrade service reliability especially with regard to fracture. Thus, the primary purpose of the present research is to provide a broad-based understanding of the

applications and consequences of selected advanced processing techniques to high performance alloys. The research ranges in scope from modeling studies of the fracture of alloys containing porosity to the high temperature deformation of rapidly solidified, dispersion-strengthened Ti alloys. Much of the research is designed so that it may also be viewed as a fundamental study of fracture utilizing engineering materials containing processing-induced defects. Significant progress has been achieved in this program during the period October 1, 1984 to September 30, 1985 in the following areas:

(1) the influence of void/pore distributions on ductile fracture,

(2) the effect of porosity on low cycle fatigue,

(3) hot isostatic pressing of metallic powders,

(4) deformation of rapidly solidified Ti alloys at elevated temperatures, and

(5) the combined effect of stress state and grain size on hydride-induced hydrogen embrittlement.

The educational experience that the above research provides to the graduate students is also significant. The following students have been supported by this program during part or all of the past fiscal year: Stephen Kampe, Ph.D. candidate; Barbara Lograsso, Ph.D. candidate; Dale Gerard, Ph.D. candidate (M.S., May 1985); Paul Magnusen, Ph.D. candidate; and Ellen Dubensky, M.S. August, 1985.

(1) Void/Pore Distributions and Ductile Fracture (with Paul Magnusen, Ph.D. candidate and Ellen Dubensky, M.S., 1985)

The presence of porosity is frequently a problem in alloys which have been cast, processed by powder metallurgy techniques, or welded. In addition, wrought alloys typically contain large intermetallic phase particles which crack and form voids at small strains when fracture of the matrix is still remote. In either instance, the deleterious influence of pore and/or

voids on strength and especially tensile ductility is well known (for example, see ref. 7). It is also well established that the severity of the effect depends on the volume fraction of pores or void-nucleating inclusions; however, factors such as the inclusion size and the degree of inclusion clustering should also influence fracture resistance. A difficulty in analyzing the influence of porosity or voiding on ductile fracture is that changes in one factor, for example the volume fraction of porosity, are usually accompanied by changes in other factors, such as the distributions of pore sizes, pore shapes, and interpore spacings. Thus, an experimental separation of the individual effects of a pore/void microstructure on strength and fracture behavior do not exist.

The above situation is not confined to experiment; it also extends to theory. Nearly all theoretical analyses of ductile fracture based on void growth and linking assume regular arrays of equi-sized holes or cavities. Only Melander has attempted to analyze the effects of a random distribution of voids.[9]

Many previous studies, both experimental and theoretical, have used through-thickness holes as a two-dimensional analog of a three-dimensional distribution voids or pores. In the present study,[10] void distributions are modelled in two dimensions as arrays of holes whose positions are predicted using a random number generator. Experiments are conducted on arrays of equi-sized fracture behavior are monitored. The study is based on two materials (1100 Al and 7075-T6 Al) of differing work-hardening rates which are tested under conditions of either plane stress or plane strain: (a) 1100-0 Al in the form of 1 mm sheet (plane stress deformation), (b) 7075-T6 Al also as 1 mm sheet and (c) 7075-T6 plate (6 mm thick) in which deformation between the holes is predominantly plane strain through the specimen thickness. The initial experimental design used is that of a 2^3 factorial in which 2 values are chosen for each of the three parameters: area fraction of

holes, hole size, and hole spacing. Specimens of the 7075 plate containing periodic arrays of holes are also tested for comparison with data from corresponding plate specimens containing random arrays. It should also be noted that the experiments do not attempt to explore a wide range of parameter values; rather they are intended as an initial study which indicates major trends. A more extensive study is currently in progress; this includes a wider range of hole area fractions and spacings as well as tests on a material with a higher strain hardening capacity (alpha brass) or possessing strain-rate hardening (low carbon steel).[11]

The experimental observations in the present study lead to the following conclusions:[10,11]

1. The value of the minimum hole spacing, S_m, which indicates the degree of clustering among holes, has a strong influence on both ductility and yield/tensile strength. If clustering is inhibited by increasing S_m, ductility as well as strength increase.
2. The diameter of the holes also has the effect that decreases in both ductility (elongation to fracture) and strength (yield and tensile strengths) occur when hole size is increased.
3. The decrease in tensile strength with increasing area fraction of holes occurs in a manner consistent with data for porous P/M alloys; see Fig. 1. However, the dependence of ductility on the area fraction of holes <u>by itself</u>, is not as pronounced as porosity-effect data from bulk specimens; see Fig. 2.
4. Hole extension in the tensile direction occurs at a rate which is linear with strain. These data also show an eventual strain-induced accelerated hole growth among some but not all of the holes in the fracture path.
5. Macroscopic observations show that, upon deformation, strain localizes along a plane of maximum shear stress between either a pair

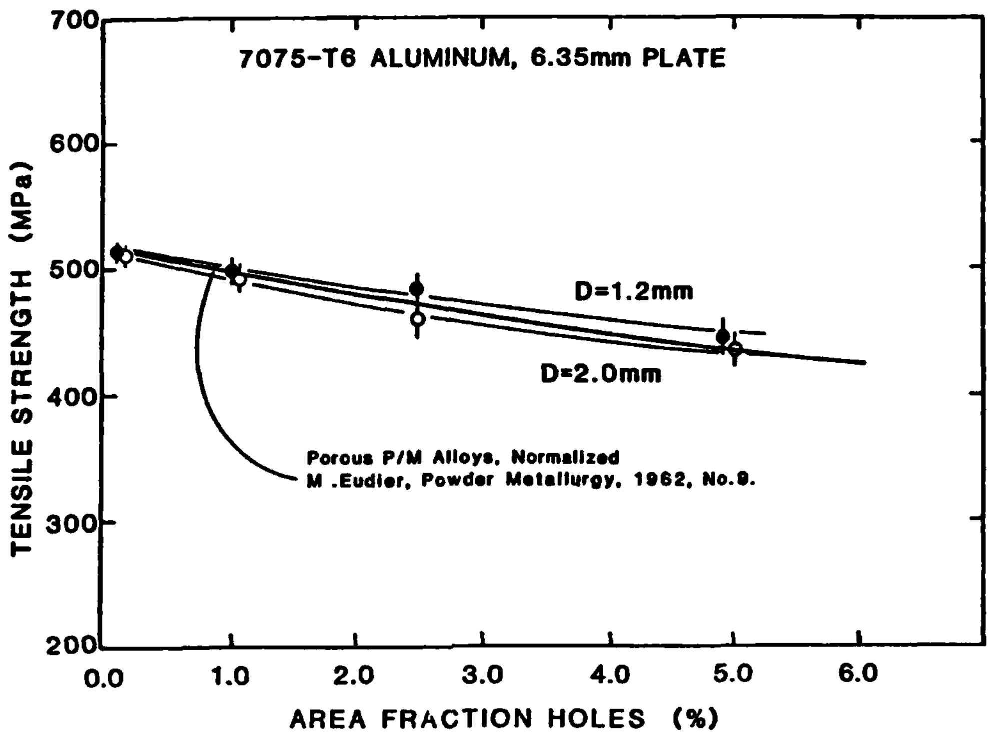

Fig. 1. The influence of the area fraction of holes on the tensile strength of 7075-T6 Al plate with either 1.2 or 2.0 mm diameter holes.

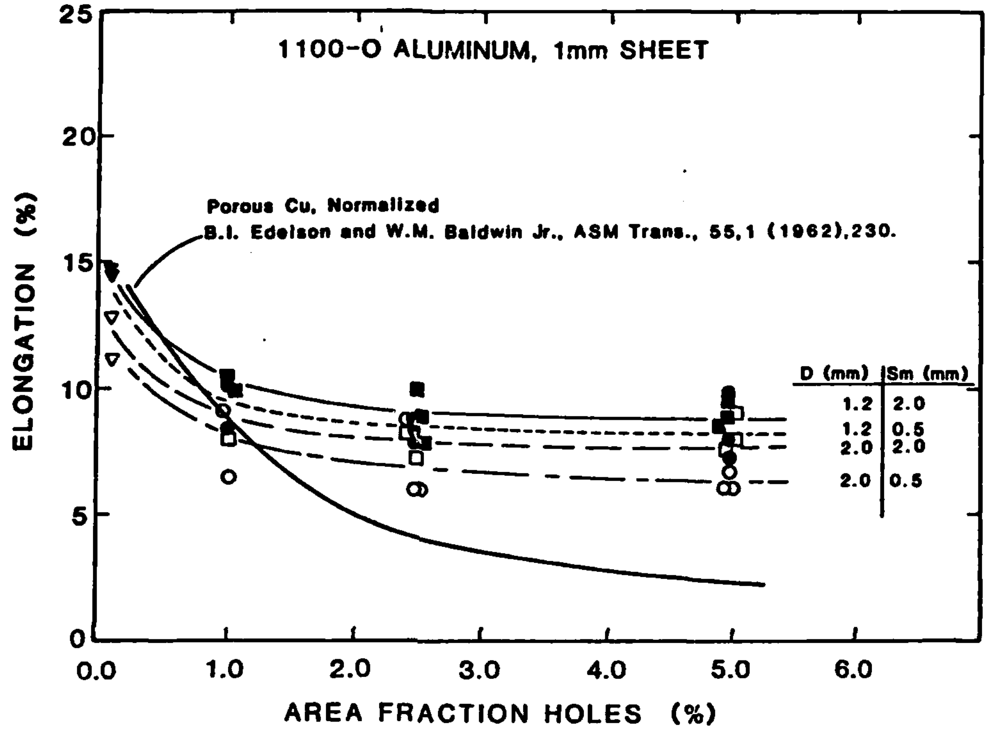

Fig. 2. The dependence of tensile elongation to fracture on the area fraction of holes with diameters of either 1.2 or 2.0 mm and minimum hole spacings of 0.5 or 2.0 mm. Data are for 1100-0 Al sheets.

or groups of holes which are both closely spaced and properly oriented with respect to the stress axis. This cluster of holes eventually forms the fracture path as the flow localization causes ligaments to fail.

6. The final fracture path for a particular array of holes depends on the stress state between the holes (or the orientation of the maximum shear stress planes). The two sheet materials tested in this study (1100-0 Al and 7075-T6 Al) showed identical fracture paths for many of the hole arrays, whereas the plate material (7075-T6 Al) usually chose a much different fracture path. The area fractions of holes on the fracture surfaces are on the average eight to ten times the original nominal area fractions.

7. The 7075 plate specimens with regular arrays of holes exhibit three to four times more ductility than those containing random arrays for the same hole size and area fraction.

The above conclusions are the basis of the following proposed sequence for the flow and fracture of materials containing voids or pores.

I. Slip initiates near individual holes (or voids/pores) as plastic zones form.

II. Flow is localized on planes of high shear stress between closely spaced and favorably oriented holes.

III. An individual ligament between two holes fractures and creates a large elliptical hole which, due to its increased size and eccentric shape, tends to further localizes flow along its major axis.

IV. A statistical problem arises as to whether or not a third hole exists in a favorable position with respect to the large elliptical cavity. If a third hole is favorably located, successive flow localization

and ligament fracture occurs, but if not, deformation of the entire material continues until another pair of holes/voids link up by ligament fracture, re-initiating the above sequence.

V. A group of holes (voids/pores) which have linked up cause a large imperfection. This in turn causes further localization and subsequently a shear instability over a much large scale and finally, the material fractures.

The above sequence is sensitive to (a) minimum spacing between holes (which strongly affects shear localization between holes), (b) hole size (which controls plastic zone size and therefore the physical scale of the shear localization possible), (c) strain hardening (which tends to diffuse localization and delay the shear instabilities) and (d) in a material containing porosity or microvoids, the area fraction of pores/voids (which affects the strain necessary to link sufficient voids/pores to create the critical imperfection).

The focus of current work is to develop a theoretical basis for predicting failure based on the plastic zone geometry around holes, the statistical probability of plastic zone overlap, the plasticity around linked-hole pairs, and on imperfection theory to describe macroscopic fracture. If the analysis is successful for equi-sized holes, it should be possible to predict theoretically the effect of a distribution of hole/void hole sizes. This would constitute a very significant advance in the understanding and prediction of ductile fracture.

(2) <u>The Influence of Porosity on Low Cycle Fatigue</u> (with Dale Gerard, Ph.D. candidate).

Our previous studies of the influence of porosity on tensile fracture [12-13], as well as the above modeling studies, indicate that porosity acts to trigger shear instabilities which localize flow within the ligaments between

closely spaced pores. Failure of individual ligaments subsequently increases the severity of strain localization process, and failure eventually occurs due to non-uniform plasticity. The <u>elastic</u> stress concentrating effects of porosity and its deleterious effects on <u>high</u> cycle stress-life fatigue behavior is well known[7,14]. Despite the potential usage of components containing porosity in <u>low</u> cycle fatigue (LCF) conditions, the effects of porosity on (LCF) behavior wherein the component is fully plastic have not been studied systematically. Only a few studies have explored specific alloy/porosity conditions[15-17]. The dependence of fracture on flow localization between pores suggests that LCF, which in itself tends to localize slip, should be quite sensitive to porosity and that this sensitivity should depend on the strain hardening and cyclic stability behavior of the matrix.

The purpose of this research is to examine in detail the fundamental mechanism(s) by which porosity interacts with cyclic deformation and affects low cycle fatigue behavior. The program will be based initially on the low cycle stress-strain response of commercially pure Ti which is being used as a model material containing different pore "microstructures" and which will be tested at two different temperatures (25 and approx. 400°C) to simulate different matrix strain-hardening characteristics under conditions of identical pore microstructure. Powder processing techniques are currently being used to obtain specimens of varying volume fraction of porosity, pore shape and (within limits of the starting powders) pore size.

Presently, specimens of the titanium are being fabricated in a multiple step process consisting of cold isostatic pressing, vacuum sintering, swaging, and resintering. Materials are being processed at (a) three levels of pore content, (b) two levels of pore sizes, and (c) rounded as well as irregular pore shape.

(3) Hot Isostatic Pressing of Metallic Powders (with Barbara Lograsso, Ph.D. candidate)

As a means of eliminating the deleterious effects of porosity, hot isostatic pressing (HIP) is being used as a means of compacting powders, rapidly solidified ribbons, and castings to full density. In the absence of fundamental studies of HIP, unnecessarily high temperatures and pressures (plus long times) are commonly used to insure full density. For starting materials (powders/ribbons) processed by rapid solidification, this has the potential of eliminating many of the advantages inherent in the starting materials. Thus we are conducting a study which seeks to establish pressure-time-temperature relationships which characterize HIP for a range of metallic powders.

The response of metal powders to the simultaneous application of temperature and pressure during hot isostatic pressing is a complex process during which particle rearrangement, plastic deformation, and creep occur. Recently, a theoretical model[18,19] has been proposed to predict densification on the basis of creep and plastic deformation mechanisms during hot isostatic pressing. For the plastic flow and creep mechanisms, the densification rates should be sensitive to the state of stress between particles; this in turn should be a function of the particle-particle surface contact area or contiquity of the powder compact.* Thus the theoretical model[18,19] is based on the isostatic compaction of monosize spheres and a relationship for the contiquity (C_m) which increases with increasing relative density according to:[20]

$$C_m = \frac{D-D_o}{4\Pi D}\,[160(D-D_o)^2 + 16] \qquad (1)$$

*The term contiquity refers to the fraction of the surface area of powder particles which are in contact with each other.

where D_o and D are the initial and final normalized densities (as normalized to fully dense) of the powder compact.

Comparison between Eq. 1 and experimentally determined values of contiquity for HIP'd monosized spherical powders of Ni, Ti, Ti-6Al-4V and 316 stainless steel is shown in Fig. 3. These data, which are obtained for a wide range of HIP conditions (34-100 MPa pressure, 700°-900°C, and 3.0×10^2 to 3.2×10^4 s), show very good agreement with Eq. 1, which is a basis for theoretical modeling of the HIP process.[18,19]

This study is also making direct comparisons between the HIP analyses and observed behavior. Fig. 4 shows good agreement between the HIP theory[18,19] and experiment observations for spherical Ti powders HIP'd at 700°C/34 MPa. Most of the other data for monosized spherical powders also show good agreement between observed and predicted densification.

Current research is focusing on the HIP behavior of (a) spherical Ti powders with a distribution of powder sizes and (b) irregular-shaped powders. Preliminary data for fine (35-44 μm) hydride-dehydride Ti powder with irregular shape is shown also in Fig. 4. Despite a very much lower initial packing density (D_I = .43 for this irregular powder but D_I = 0.64 for the spherical particles), the irregular powder appears to densify rapidly and in a manner similar to that of the spherical powder. This result agrees with the concept that at least at high densities (>0.90) densification of irregular powders should be similar to that of spherical powders.[21] A wider range of experimental data are currently being obtained for irregular powders.

(4) <u>Deformation of Rapidly Solidified Titanium Alloys at High Temperatures</u>

(with Steve Kampe, Ph.D. candidate)

Rapid solidification (RS) represents a novel processing strategy which is currently being extensively explored as a means to increase the service

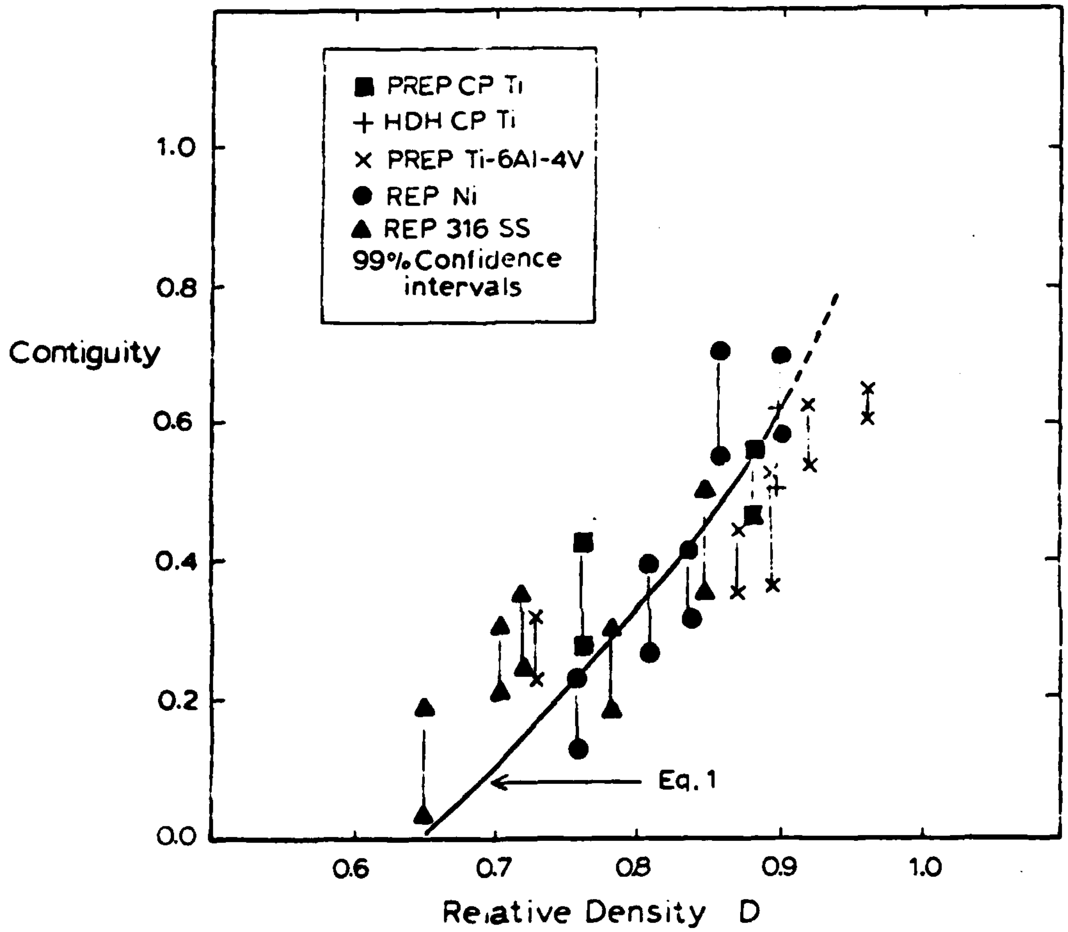

Fig. 3. The observed and predicted dependence of interparticle contiquity C_m as a function of the relative density of powder compacts subjected to HIP.

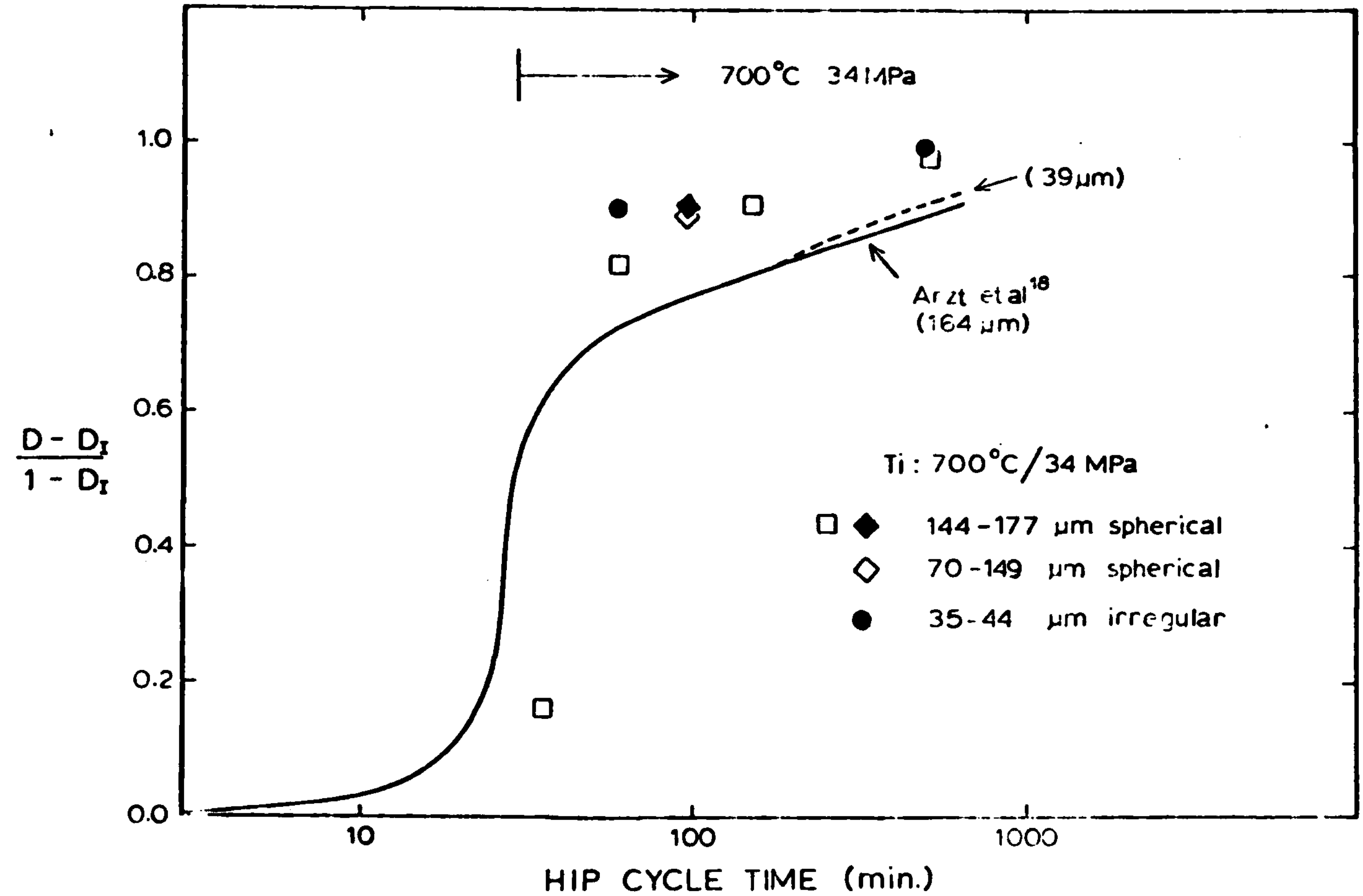

Fig. 4. Normalized densities of HIP'd metal powders as a function of HIP cycle time at 700°C/34 MPa.

temperature capability of titanium-base alloys for high performance applications. Extending the use of titanium to higher temperatures (e.g. 700°C) would result in very large improvements in aircraft design efficiency as a result of the displacement of various heavier (more dense) nickel- and iron-base alloys currently employed in advanced gas turbine engines.

Similar to the development of oxide-dispersion strengthening of nickel-base alloys, considerable alloy development efforts have been directed to the disperson strengthening of a Ti alloy using rapid solidification (RS) processing. Despite promising data obtained from RS Ti alloys in the form of ribbons, flakes, or laser-melted surfaces[4,22,23], there has been no thorough study of the high temperature deformation and fracture behavior of RS Ti alloys in bulk form. Published research is confined to but a few tests[24], and these indicate that while some strengthening occurs due to dispersion strengthening, the retention of strengthening to high temperatures may not be as good as expected. This appears to be the case for several experimental alloys being developed.[25]

The purpose of this proposed project is to study in a fundamental manner the influence of temperature on the deformation and fracture of systems which model potential high temperature RS Ti alloys. The alloys, which are based on erbium additions, rely on oxide-dispersion strengthening combined with other sources of strengthening, such as solid solution hardening, to achieve good creep resistance. The model alloys consist of oxides dispersed in pure alpha-phase matrix (CP Ti), solid-solution strengthened alpha-phase matrix (Ti-6Al), and age-hardened plus solid solution strengthened alpha-phase matrix (Ti-10Al and Ti-12Al).

To date, 200 grams each of Ti-2Er and Ti-6Al-2Er have been received from Dr. Sung Whang. With the assistance of personnel at General Electric Research Labs (Schenectady), these alloys have been HIPped and hot-extruded into bulk

form, yielding a fully dense, homogeneous, fine grained microstructure as shown in Fig. 5. These transmission electron micrographs reveal the following observations: (a) a very fine grain size ($d \leq 5$nm), (b) the presence of the oxide dispersion, with dispersoids residing both inter- and intragranularly, (c) the broad size distribution of these dispersoids; preliminary measurements indicate diameters ranging from 5 nm to 500 nm, and (d) the occurrence of regions of both high and low density of dispersoids.

Moderately slow strain rate compression tests were used to assess the mechanical performance of the RS dispersoid-containing alloys. Parallel tests were conducted on conventional, powder fabricated C.P. titanium for comparison. Elevated temperature testing (300, 600, 700, and 775°C) was performed on an Instron testing machine equipped with specially designed compression fixtures which prevent specimen buckling. Flowing, high purity argon was used within a sealed atmosphere tube to prevent excessive oxidation of the titanium during testing.

Fig. 6 illustrates the results of the high temperature testing of the CP Ti, Ti-2Er, and Ti-6Al-2Er, represented on a Dorn-type plot, i.e. log of near-steady state creep rate ($\dot{\varepsilon}_{ss}/D$) versus log of steady state creep stress (σ_{ss}/E), where D and E are the temperature compensated diffusion coefficient and elastic modulus, respectively. These data show a definite positive influence of the dispersoids on the creep resistance of these alloys at higher stresses (i.e. lower temperatures and higher strain rates), but a decrease in strengthening at lower stresses (i.e. higher temperatures and lower strain rates). The data for all alloys tend to approach that of the unstrengthened CP titanium under the extreme conditions of high temperature (i.e. 775°C) and low strain rates ($\dot{\varepsilon} \leq 2.5 \times 10^{-4}$ s^{-1}).

The degradation of creep resistance at higher temperatures for these fine grained dispersion strengthened alloys strongly suggests that grain boundary

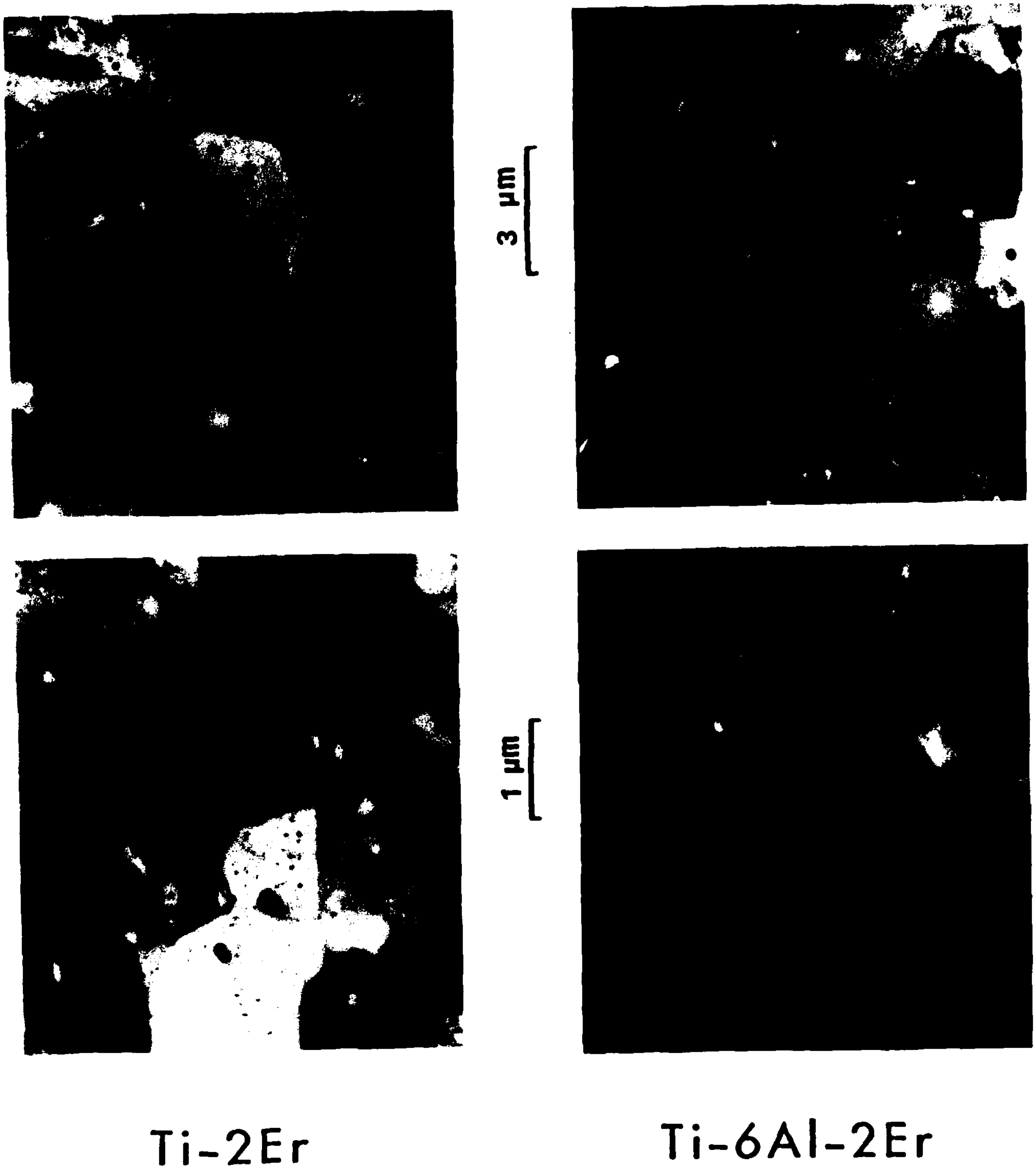

Fig. 5. TEM micrographs of the rapidly solidified Ti-2Er and Ti-6Al-2Er alloys showing microstructure following hot-extrusion and a 775°C / 2 hr anneal.

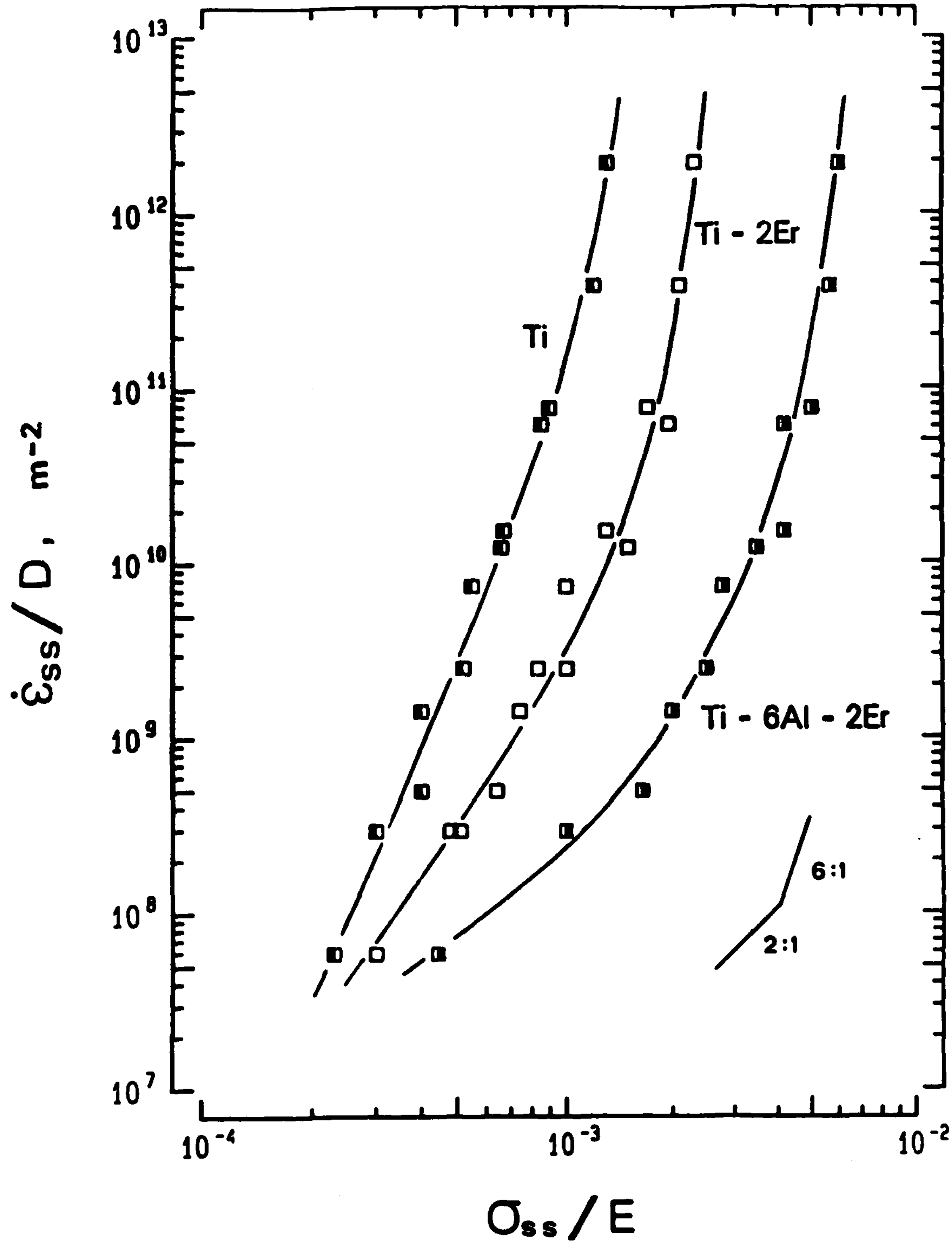

Fig. 6. A Dorn-type plot illustrating the results of the high temperature (600, 700, 775°C), slow strain-rate compression testing for CP Ti, Ti-2Er, and Ti-6Al-2Er. The slope of these curves correspond to the Dorn equation stress exponent, n.

sliding may be a predominant deformation mechanism under these extreme conditions. This is further supported by the gradual decrease of the Dorn equation stress exponent, n (as defined by the slope of the curves of Fig. 6), from a value exceeding 6 at lower temperatures to a value approaching 1 at higher temperatures. The possibility of grain boundary sliding is currently being investigated using microscopy.

Future research will involve (1) a more extensive structural characterization of the RS alloys, including dispersoid size and areal distribution. (2) continuation of the mechanical testing of RS alloys, including RS alloys of higher aluminum contents (Ti-10Al-2Er and Ti-12Al-2Er) recently obtained from General Electric, and (3) a closer examination at the origin of the property degradation at higher temperatures, with specific interest in the possible role of grain boundary sliding as the controlling deformation mechanism. In this regard, heat treatments designed to grow larger grains in the RS alloys are planned.

(5) <u>The Combined Effect of Stress State and Grain Size on Hydrogen Embrittlement of Titanium</u> (with Dale Gerard, M.S. Thesis, May, 1985)

Commercially pure titanium is very resistant to embrittlement due to hydrogen when tested in the form of fine-grained specimens at low-to-moderate strain rates in uniaxial tension.[26-28] It is well known that Ti becomes susceptible to hydrogen embrittlement in the presence of a notch, at low temperatures or high strain rates, or large grain sizes.[26-31] As a previous part of this program, we have also shown a sensitivity to stress state with Ti sheets exhibiting pronounced hydrogen embrittlement in equibiaxial tension even though the loss of ductility in uniaxial tension is negligible.[32,33] The purpose of this small project was to explore the combined effects of two of the above factors, grain size and stress state, on the hydrogen embrittlement of Ti.[34]

The combined influence of grain size and stress state on the hydrogen embrittlement of Ti sheet is shown in Fig. 7. Fig. 7 shows that there is very little, if any, effect of grain size on the ratio of fracture strain in equibiaxial tension to that in uniaxial tension for the uncharged Ti. In contrast, the loss of ductility in equibiaxial tension for the hydrogen-charged sheets is much more pronounced for the coarse-grain material than in the finer grained counterparts. Thus, the data in Fig. 7 indicate that the hydrogen embrittlement of Ti sheet at room temperature is most severe in coarse-grain material when subjected to equibiaxial tension.

That increasing either grain size (in uniaxial tension or biaxiality of stress state (at constant grain size) increases the severity of hydrogen embrittlement in hydride-forming alloys is known.[27,28,32,33] Thus it is not entirely surprising that the stress-state effect is itself sensitive to grain size. The cause of this effect may be understood in the context of the previous studies, metallographic/fractographic observations, and the void nucleation/void link-up steps of the ductile fracture process. Grain-size effects in uniaxial tension have been previously attributed to an increased hydride plate size associated with the coarser grains.[27,28] This is consistent with present observations of <100 μm hydrides in the coarse-grain sheets but <40 μm hydrides in the fine-grain material. The combination of large hydrides and large normal stresses required to deform the plastically anisotropic sheets in equibiaxial tension should decrease the strain to fracture of the hydrides, thus enhancing void nucleation at smaller strains. In addition, the presence of large, interconnected hydrides also assist void link-up by providing paths of easy fracture along the flat hydride faces,[33] especially under the multidirectionality of the two major principal stresses in equibiaxial tension. This effect is apparent on the fracture surfaces of the coarse-grain material which show faceting due to void link-up along the

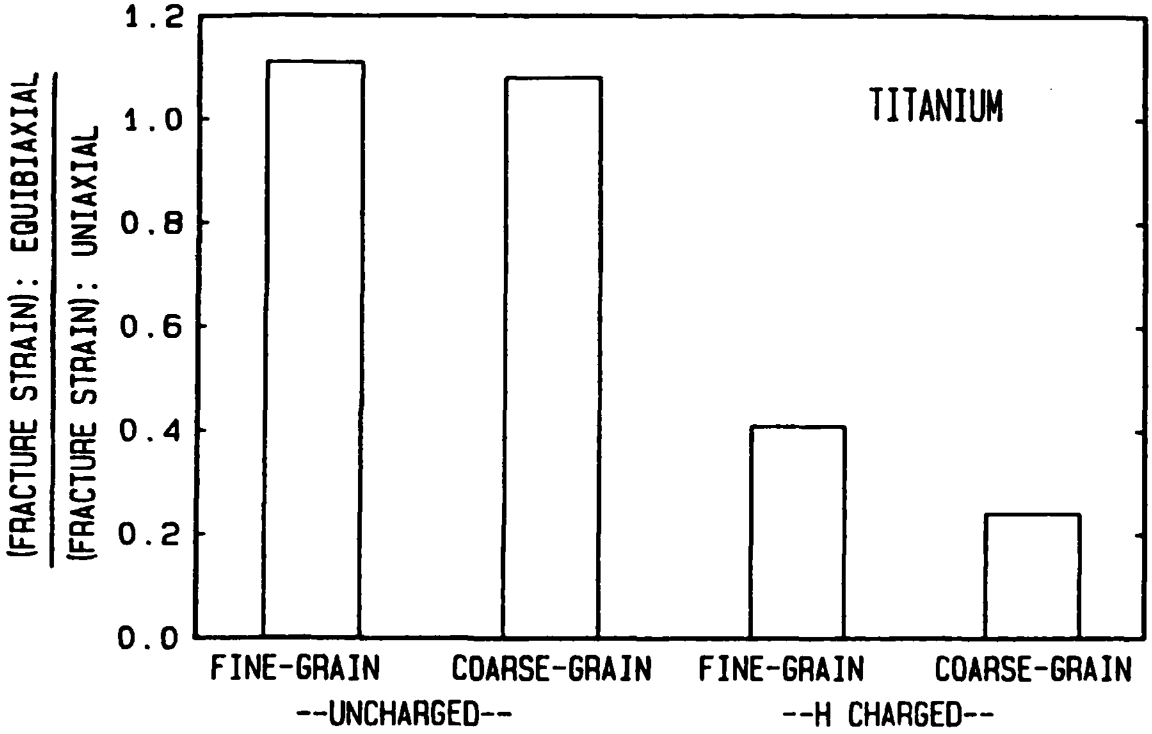

Fig. 7. The ratio of Hill equivalent strain to fracture in equibiaxial tension to that in uniaxial tension for Ti with either 0.015 or 0.030mm grain size and 25 or 1850 wt ppm H.

hydride plate faces in both uniaxial and equibiaxial tension. Such faceting is most pronounced in the coarse-grain material tested in equibiaxial tension, indicating the enhancement of void link-up when both large, interconnected hydrides and a multidirectional tensile stress state are present.

In view of the above, we may summarize. Increasing grain size increases the susceptibility of Ti (and probably other hydride-forming alloys) to hydrogen embrittlement at high degrees of stress biaxiality/triaxiality. This effect appears to be a consequence of an enhancement of both void nucleation (due to hydride fracture) and void link-up at large grain sizes/biaxial stresses. Void nucleation should be enhanced as the large grains create conditions for large hydrides[27,28] which in turn fracture and form voids at smaller strains at the large normal stresses required to deform plastically anisotropic (R and P>1) sheets in equibiaxial tension.[33] Void link-up should also be enhanced as the large interconnected and plate-like hydrides create paths for especially easy void link-up when subjected to the multidirectional major principal stresses in equibiaxial tension. These effects should be even more pronounced under triaxial states of stress, such as near notches or cracks, provided that the stress state is sufficiently long range to encompass several large grains/hydrides, thus permitting enhancement of both void nucleation and link-up.

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